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Fabrication of ZnO Thin Film through Chemical Preparations

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Abstract

Zinc oxide (ZnO) is a compound that has unique physical and chemical properties. It has a direct band gap at 3.4 eV (without dopant), a high bonding energy (60 meV), and a high thermal and mechanical stability at room temperature. Thus, ZnO thin film can be suitably applied in many fields, and it also has many functions such as UV light emitters, hydrophobic coating, transparent thin film in electronic devices, piezoelectric material, transducers, gas-sensing, and a transparent conductive oxide (TCO) layer in thin film solar cells. ZnO thin film could be prepared by many chemical preparations such as chemical bath deposition (CBD), chemical vapor deposition (CVD), sol-gel spin coating, doctor blade, printing deposition, and electrochemical deposition (ED). This chemical process is a low-cost, simple, and easy preparation process to be adjusted or doped by other elements.

Keywords: zinc oxide, thin film, chemical preparations, TCO, solar cells

1. Introduction

Zinc oxide (ZnO) is a II-VI semiconductor group which has a wurtzite as the most stable compound structure and has unique physical and chemical properties. In physical properties, ZnO has a high thermal stability, which starts to melt and boil at 1975°C; in optical properties, it has a high transparency and a direct band gap at 3.4 eV, which can be tuned by doping several elements such as Al, Ga, or In [1]. ZnO also has a low resistivity, which is about $1-2 \times 10^{-4} \Omega\text{cm}$ [2], a high thermal stability, a high electrochemical coupling coefficient, and a high bonding energy (60 meV) [1]. Due to all these unique properties, ZnO becomes a promising material, which can be applied in many fields as a sensor, converter, catalyst, and even as an important part in solar cells. This study aims to review ZnO as a transparent conductive oxide (TCO) in solar cells.

2. Zinc oxide as thin film in solar cells

In solar cells, ZnO plays important roles in collecting the energy from sunlight in various solar cells such as silicone-based solar cell (first-generation solar cell), thin film (second-generation), organic, multijunction, dye-sensitized (third-generation), hybrid and perovskite solar cells (fourth generation). In the first to the third generation of solar cell, ZnO plays a role as transparent conductive oxide (TCO), except in organic solar cell in which ZnO acts as a junction for exciton separation [3].

Transparent conductive oxide (TCO) is very important for harvesting electricity from sunlight and can improve solar cell efficiency. This layer is used to place on top of thin film solar cells and has a high transmittance (greater than 80% in the visible spectrum area) but has a low resistivity (less than $10^{-4} \Omega\text{cm}$). These properties will minimize the probability of the received photon absorbed by the layers before the absorber layer or reflected back. **Table 1** shows several properties of ZnO as TCO on solar cells.

In addition, TCO should have a higher band gap than the absorber layer and the buffer layer, approximately 3.2 eV. If all of the layers are stacked together, they will make a gradation of band gap energy from low band gap energy (absorber layer) to the high energy (the top contact). This gradation can help electrons of the absorbed photons in the absorber layer to move

Property	Value
Lattice parameters at 300 K	
a_0	0.32495 nm
c_0	0.52069 nm
a_0/c_0	1.602 (ideal hexagonal structure shows 1.633)
u	0.345
Density	5.606 g/cm ³
Stable phase at 300 K	Wurtzite
Melting point	1975 °C
Thermal conductivity	0.6, 1–1.2
Linear expansion coefficient(/°C)	a_0 : 6.5×10^{-6} c_0 : 3.0×10^{-6}
Static dielectric constant	8.656
Refractive index	2.008, 2.029
Energy gap	3.4 eV, direct
Intrinsic carrier concentration	$<10^6 \text{ cm}^{-3}$ (max n-type doping $>10^{20} \text{ cm}^{-3}$ electrons; max p-type doping $<10^{17} \text{ cm}^{-3}$ holes
Exciton binding energy	60 meV
Electron effective mass	0.24
Electron Hall mobility at 300 K for low n-type conductivity	200 cm ² /V s
Hole effective mass	0.59
Hole Hall mobility at 300 K for low p-type conductivity	5–50 cm ² /V s

Table 1. Properties of ZnO [1].

past the other layers to the top layer. On the other hand, the holes move to the back-contact as a hole summation in solar cells. Since the electrons and holes move to the opposite side, this makes a difference potential on either side and produces electricity. If the band gap gradation is not sequentially arranged, it will disturb electron and hole movement. As a consequence, the electricity will not appear. **Figure 1** shows illustrations of electrons and holes, movement through several layers with different band gaps in the CIGSe (CuInGeSe_4) solar cell.

Besides its physical properties, ZnO is a common material as TCO for thin film solar cell because this material is non-toxic and abundant. However, ZnO still lacks in optical and electrical (opto-electrical) properties. Thus, some of elements or molecules have been successfully developed to improve its optoelectrical properties such as Al, Ga, B, In, Y, Sc, F, V, Si, Ge, Ti, Zr, Hf, In_2O_3 and SnO_2 [6]. Some of elements provide a significant change in optical and electrical properties of ZnO. **Table 2** shows some changes of ZnO optical and electrical properties as TCO after being doped by some elements.

ZnO thin film could be prepared by physical or vacuum methods such as radio frequency (RF) magnetron sputtering or direct current (DC) sputtering, pulsed laser deposition (PLD), and e-beam evaporation [12–15]. However, all of these methods need particular vacuum equipments and investment. Contrary from physical or vacuum methods, chemical methods are well known as a low-cost method because it does not require particular equipment or expensive investment. Thus, chemical process becomes a promising and low-cost process.

There are several processes for depositing ZnO by using chemical process, such as chemical bath deposition (CBD)/dip coating, chemical vapor deposition (CVD)/spray pyrolysis, sol-gel spin coating, doctor blade coating, printing deposition, and electrochemical deposition. Even though there are various deposition techniques, the steps of each technique are the same. The first step is preparing the precursor which can be in a solution, sol-gel, colloidal, or even in a dispersion precursor form. The second step is depositing using various techniques, and the last step is drying or heat treatment to remove the solvent and to develop the ZnO thin film. Common steps in the preparation of ZnO by chemical process are shown in **Figure 2**.

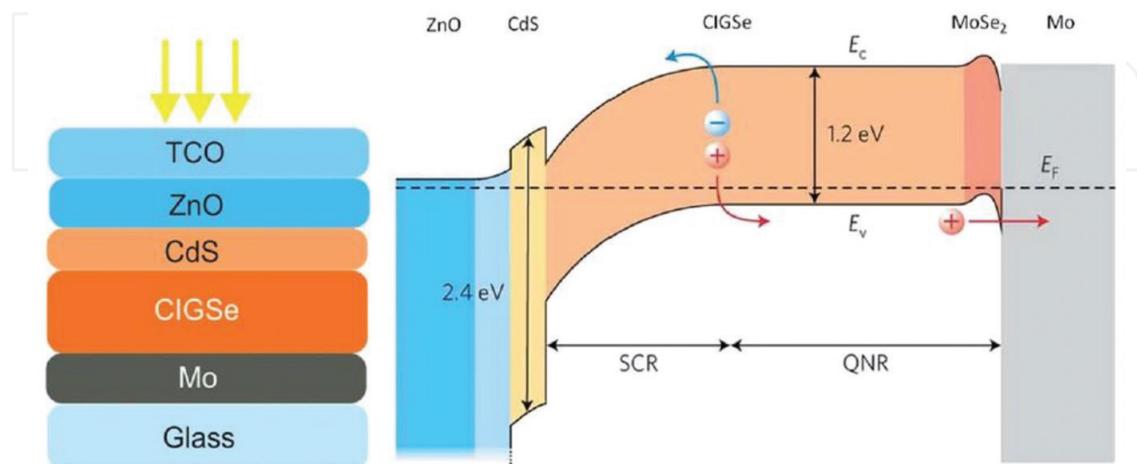


Figure 1. A schematic layer structure of a classical CIGSe solar cell [4] and a schematic band profile of a typical CIGSe solar cell under zero bias voltage condition [5].

Dopants	Optimum content in target (%)	Thickness (nm)	Resistivity (Ωcm)	Transmittance (%)	References
Al	2	500	4.5×10^{-4}	88	[7]
Ga	5	200	8.12×10^{-4}	>90	[8]
In	40	>1000	4.02×10^{-4}	>85	[9]
F	2	200	4.83×10^{-4}	>90	[10]
Si	2	~150	6.2×10^{-4}	~80	[11]

Table 2. Properties of ZnO films with different dopants.

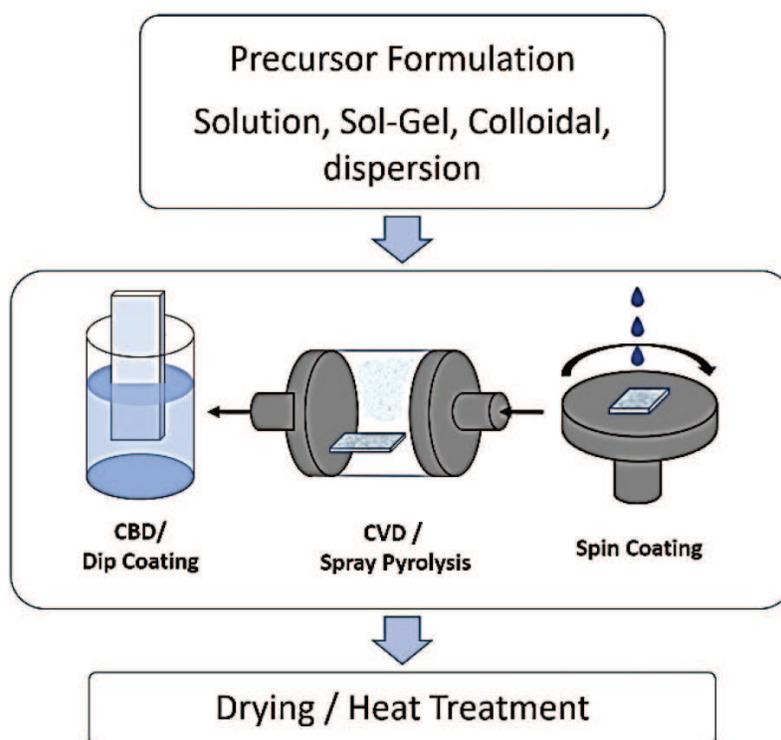


Figure 2. A common preparation step to fabricate ZnO thin film by chemical processes.

In the chemical preparation process, to adjust ZnO thin films into p or n semiconductor, it is done by adding some particular element of salt such as aluminum salt or boron salt into the precursor mixture.

Some of experiments only need a single step of heat treatment to develop the ZnO thin film, but in spin-coating method, several repetition steps of coating and heat treatment are required in order to get the desired thickness, and this repetition step is called as preheat treatment. Contrary with the spin coating, chemical vapor deposition (CVD) or spray pyrolysis method may not have specific drying or heat treatment steps because in this method, the heat treatment is done simultaneously with deposition. To adjust the thickness in CVD or spray pyrolysis, it is adjusted by the deposition time.

3. Chemical deposition methods

3.1. Chemical bath deposition/dip coating

Chemical bath deposition (CBD) or dip coating is the simplest and low-cost deposition method because this method does not need expensive and special equipment. In addition, it also has a simple deposition principle. The arrangement of equipment is shown in **Figure 3**.

At the precursor preparation, zinc oxide is prepared from zinc-salt compound such as zinc acetate dihydrate, zinc nitrate, zinc chloride, and zinc sulfate [17–21]. After these kinds of zinc salts were dissolved in the solution, zinc in the salt becomes a cation which can react with anion from basic compound, as well as form a seed of nuclei which adhere on the substrate. Besides that, zinc cation also reacts with other compounds in the precursor mixture such as surfactants which act as a binder and form zinc complex compound or in order to get desire properties of the mixture such as viscosity or homogeneity. **Table 3** shows various zinc salts and other compounds as precursor mixture.

Further, this seed of nuclei will develop as zinc-salt precipitation which adheres on the substrate, and after drying or heat treatment under various atmospheres, it becomes oxidized and forms a zinc oxide as thin film. The schematic of this preparation is shown in **Figure 4**.

This is the reaction mechanism of ZnO thin film fabrication.

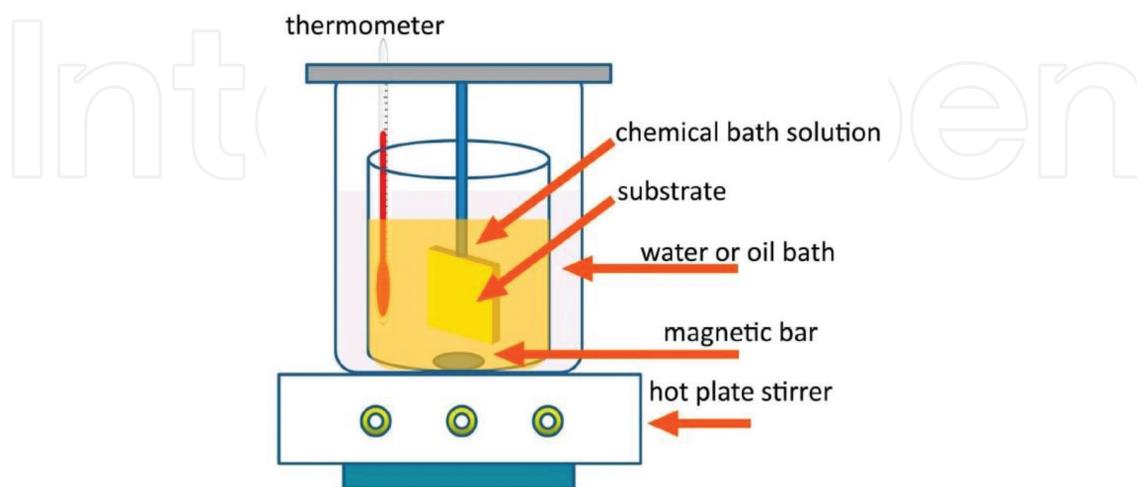
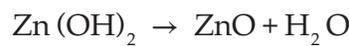
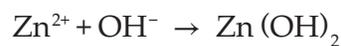
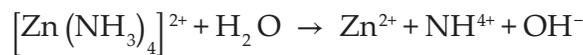


Figure 3. The arrangement of CBD equipment [16].

Zinc salt	Other additive compounds	References
$\text{Zn}(\text{CH}_3\text{COO})_2$	2-methoxyethanol, mono-ethanolamine	[17]
$\text{Zn}(\text{NO}_3)_2$	NaOH , sodium n-dodecyl sulfate, triethanolamine	[18]
ZnCl_2	NH_4OH , hexadecyl(trimethyl)azanium bromide	[19]
ZnSO_4	NH_4OH , NH_4HCO_3	[20]

Table 3. Various zinc salts and other compounds as precursor mixture.

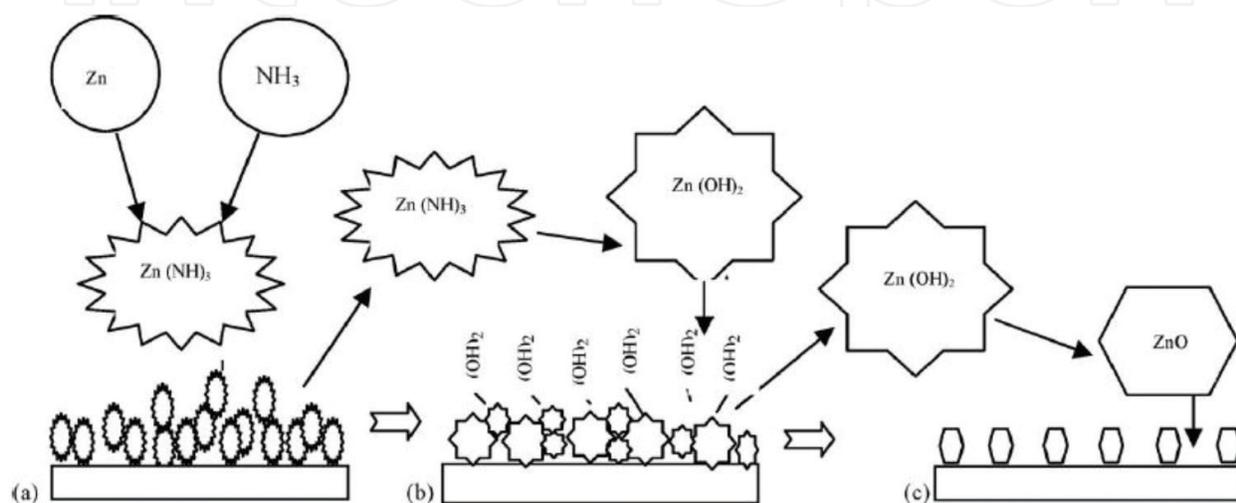


Figure 4. Mechanism of ZnO thin film growth [21].

Temperature (°C)	Duration (hour)	Atmosphere	References
100	1	Air	[23]
300	0.5	Oxygen	[24]
300–600	1	Air	[25]
400–800	1	Air	[26]

Table 4. Various annealing temperatures and atmospheres/heat treatment step.

At drying or heat treatment step, the heat treatment can be done at various temperatures and atmospheres. These various processes have the effect to the shape, morphology, optical, and electrical properties of zinc oxide thin film [22]. **Table 4** shows various annealing temperatures and atmospheres or heat treatment step.

In addition to these, the concentration of precursor of zinc salt as zinc source and other additive concentrations also give significant role to ZnO thin film [22]. The effect of zinc and surfactant concentrations can be seen in **Figure 4**. The increase of zinc and surfactant concentration shows an increase of ZnO thin film crystallinity. Besides crystallinity, the effect of

the increase of the concentration also affects the reaction rate of nucleation between zinc and anion, in which it becomes faster. However, besides concentration, the reaction rate is also affected by the temperature reaction.

3.2. Chemical vapor deposition/spray pyrolysis

The preparation of chemical vapor deposition (CVD) or spray pyrolysis is similar with CBD. The zinc precursor must be solved in a solution and has to be evaporated. Evaporation of precursor can be done by several ways. First, zinc precursor must be easy to evaporate or in other words it should be a volatile precursor. Second, by using high temperature, the zinc ingot as precursor can evaporate, and the last step is by using devices such as an atomizer or an ultrasonic transducer that can atomize the precursor or make a mist of it. The arrangement of CVD equipment is shown in **Figure 5**.

In **Figure 5**, zinc precursor solution has changed into mist by ultrasonic apparatus and carried by inert gas into the heat furnace chamber with a substrate placed inside the chamber as a target to develop the ZnO thin film. The temperatures of CVD in several experiments are shown in **Table 5**.

After the precursor mist or vapor is carried and arrived at the heater chamber, some of the zinc precursor particles undergo a reaction with a molecule that contains oxygen in the chamber and forms a larger molecule. Then, this molecule is attached on to the substrate. Due to the high temperature or enough energy, this molecule decomposes into zinc that reacts with the

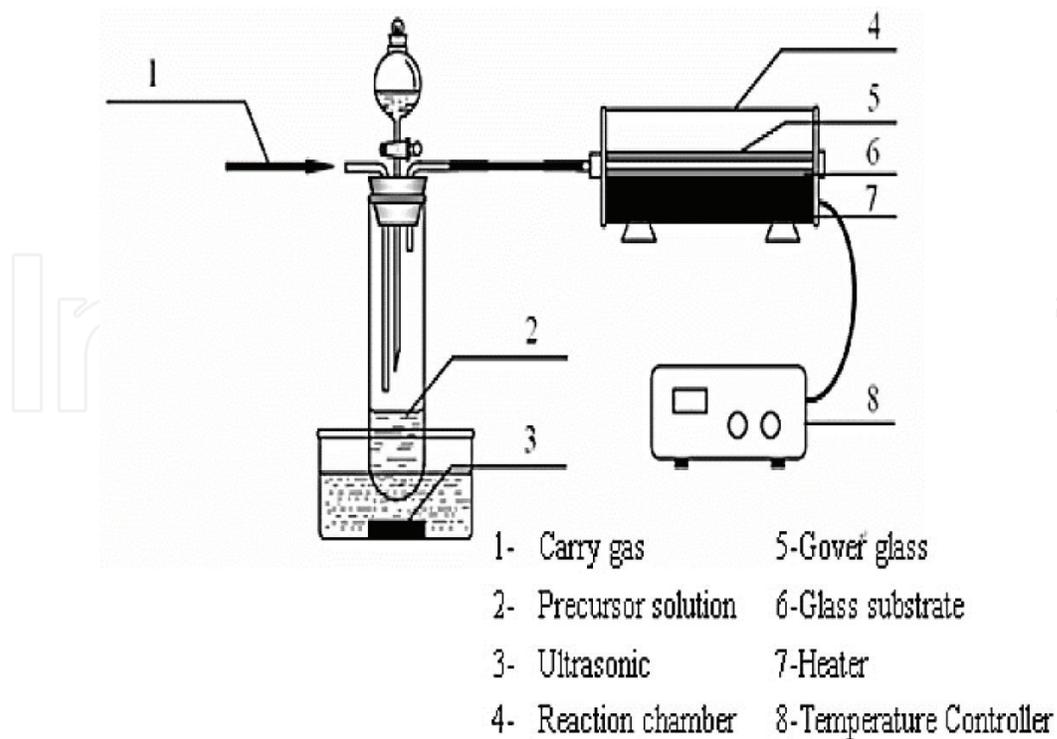


Figure 5. CVD using the ultrasonic transducer to produce a precursor mist [27].

Temperature (°C)	Zinc source	Atmosphere	References
90	Zinc acetate	O ₂ , N ₂ O, H ₂ O	[28]
135–235	Diethylzinc	He-Diborane 2%	[29]
120	Dimethylzinc	O ₂	[30]
300–375	Zinc acetate	N ₂ , O ₃	[31]

Table 5. Several ZnO thin film CVD condition.

oxygen and then forms a zinc oxide behind. The solvent is evaporated and leaves the chamber with the carrier gas, and at the same time, zinc oxide gets developed as zinc oxide thin film. Or the reaction mechanism could undergo another possibility, when zinc precursor is arrived at the chamber, and it will attach on the substrate and then undergo a reaction or bonding with another molecule which contains oxygen. Due to the high temperature and enough energy to decompose, the zinc precursor molecule and oxygen containing molecule react to the zinc oxide and release some decomposed solvents, which get carried out with gas, and finally the zinc oxide molecule becomes zinc oxide thin film. This schematic is shown in **Figure 6**.

3.3. Spin coating

Spin-coating method uses the energy of the substrate rotation to remove excess or unattached precursor and flatten the thin film. While the precursor drops on the substrate, it will attach on the substrate but the spread is uneven. When the substrate starts to spin, the precursor will spread along the substrate surface due to the centripetal force. A high spin velocity means a high centripetal force to remove the excess of precursor from the substrate. The amount of precursor in the substrate depends on the spin velocity and the precursor's ability to adhere or attach on the substrate, which is usually called as adhesivity or wet ability. It makes the precursor to attach strongly on the substrate. Besides that, the viscosity also has a role on it. If the precursor has a high viscosity, it will slowly move due to the centripetal force. Consequently, it takes a longer spinning time or a higher spin velocity. The schematic of the spin coater is shown in **Figure 7**.

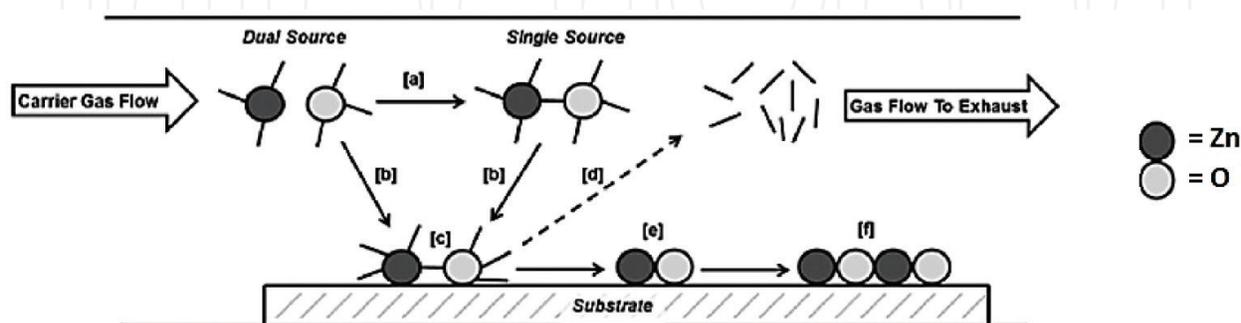


Figure 6. ZnO thin film fabrication. Mechanism with CVD [32].

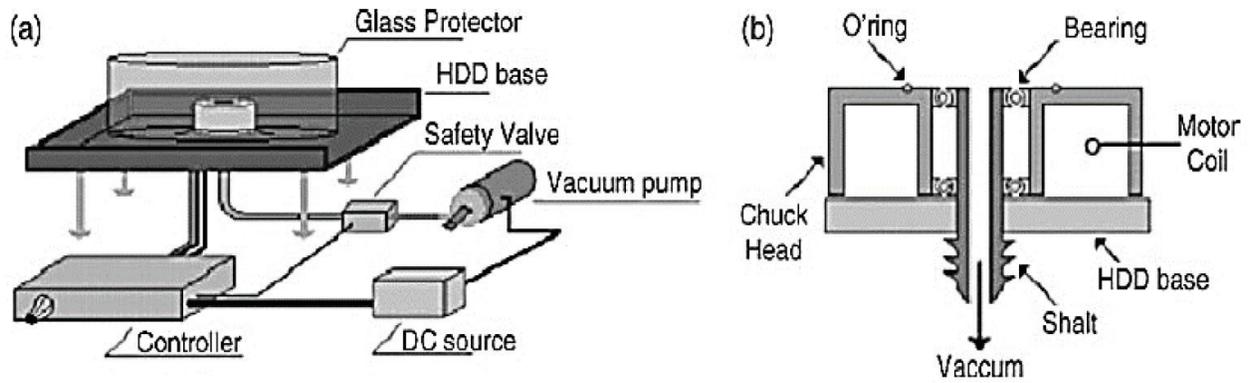


Figure 7. Schematic representation of a home-built spin coater (a) and details of the rotor (b) [33].

After the precursor is attached on the substrate, the next stage is drying stage or evaporating the solvent. One thing that should be considered is that the evaporation of the solvent is affected by the viscosity of the precursor. Thus, it is really important to find out the optimum viscosity of the precursor. A high viscosity means difficult to spread well on the substrate surface, and it means that there is a high surface tension which makes it evaporate slowly. The evaporation of the precursor's solvent is called as preheat treatment. Once the solvent evaporates, it will leave the zinc particle on the substrate and makes the layer thinner. In this method, the desired thickness cannot be achieved by a single process. It should be done in several preheat treatment stages to get the desired thickness. After the preheat treatment, the next stage is to fabricate the zinc oxide from the attached zinc on the substrate by oxidizing heating process. The schematic of alignment process on spin coating is shown in Figure 8.

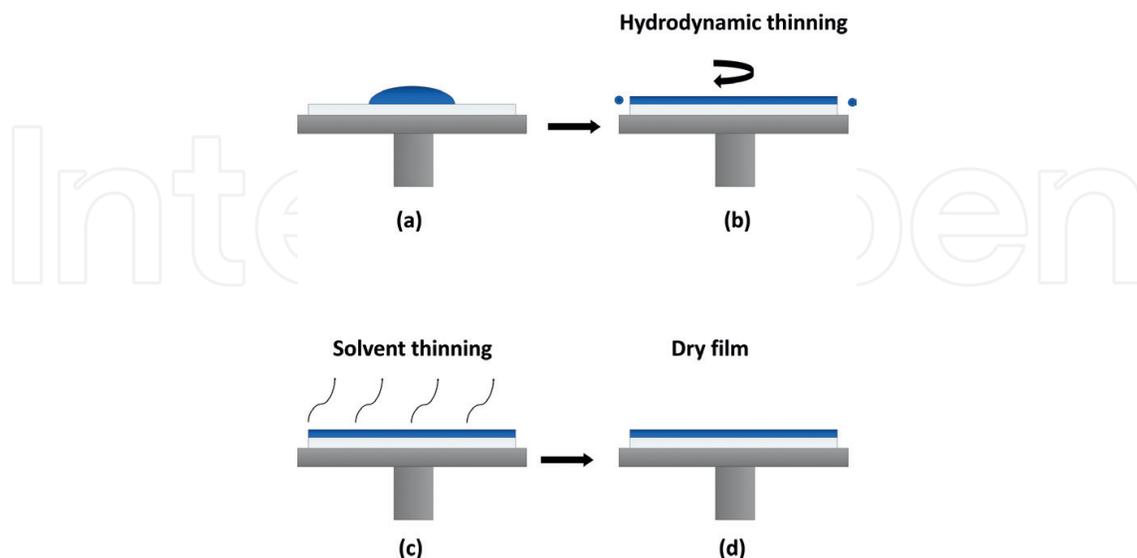


Figure 8. Schematic of the four stages of spin coating (a) Deposition, (b) Spin up, (c) Spin off, (d) Drying [34].

Precursor's form	Zinc source	Other compounds	Mixture condition	References
Sol-gel	Zinc acetate dihydrate	2-methoxyethanol, monoethanolamine	Stirring for 30 min at 60°C, aged for 72 h at room temperature	[35]
Solution	Zinc acetate dihydrate	2-methoxyethanol, monoethanolamine	Stirred at 65°C for 90 min	[36]
Solution	Zinc acetate dihydrate	Ethanol, NH ₄ OH	pH 10, room temperature	[37]
Solution	Zinc acetate dihydrate	Ethanol, acetylacetone	Room temperature	[38]

Table 6. Precursor conditions.

Spin condition	Preheat condition	Heat treatment condition	Atmosphere	References
2000 rpm for 30 s	200°C, 10 s	400°C for 1 h	—	[35]
3000 rpm for 30 s	260°C, 10 min	400°C for 3 h	Oxygen	[36]
700 rpm for 10 s	100°C for 10 min	500°C for 2 h	Air	[37]
2000 rpm for 10 s	Room temperature, 3 min	500°C for 2 h	Oxygen	[38]

Table 7. Heat treatment condition.

The precursor solution in spin-coating method is different from the chemical bath deposition and chemical vapor deposition. Precursor preparation in spin coating is more varied. It is not only a solution precursor but also a sol-gel or a colloidal form. Additionally, in this method, the quality on the thin film is affected not only by the concentration of zinc source and other additives but also by the rotation speed, time of spin coating, viscosity, and adhesivity of the precursor solution as mentioned earlier. **Table 6** shows several conditions of ZnO spin coating.

Besides that, another thing that brings this method unique lies on the preheat treatment step. The function of this step is to make the coated film dried and attached on the substrate. The repetition of this step is to get a desired thickness. **Table 7** shows the condition of several pre-heat treatments and heat treatments.

4. Conclusions

In the thin film solar cells, zinc oxide (ZnO) layer acts as a transparent conductive oxide (TCO), which is an important part of solar cells. This thin film can be fabricated by various chemical processes as alternative of vacuum process, where the production is economically cost-effective but still has the same function and properties as vacuum methods.

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